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RESEARCH MEMORANDUM

COMPATIBILITY OF PENTABORANE WITH MATERIALS USED FOR SEALS, GASKETS, AND CONSTRUCTION

By Samuel Kaye and Frank V. Sordyl

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

NATIONAL ADVISORY COMMITTEE
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SUMMARY

The compatibility of pentaborane with aluminum foil, 24S-T and 17S-T aluminum, and 50-50 tin-lead solder was determined in the range -30° to -50° F for 7 hours, at 90° F for $6\frac{1}{2}$ hours, and at 70° F for 28 days. Results were evaluated by determination of the loss in weight after the test period, metallographic inspection, and comparison with blanks carried through test conditions without exposure to the liquid.

The compatibility of pentaborane with Teflon and Kel-F polymers, natural and synthetic rubbers, asbestos and cellulose fibers, and a graphite packing was also examined after 7 days immersion at 70° F. The evaluation of results was made by visual inspection and physical tests of dimensional stability, tensile strength, elasticity, and gain or loss of weight.

The metals and fibers were unattacked and unchanged during the tests, except for the possibility that the fibers might have had low wet test strengths while actually immersed in the liquid. Teflon and Kel-F showed no change in weight or dimensions, and appeared immune to reaction with pentaborane. The rubbers generally lost about 90 percent of their tensile strength, absorbed liquid equal to about 30 percent of their weight, and showed a general and severe deterioration in structure. Loss of weight of the graphite O-ring indicated that it resisted attack, but that binders in such compositions might be leached out by pentaborane.

CO-1

INTRODUCTION

An investigation was undertaken at the NACA Lewis laboratory to determine the compatibility of various materials used for seals, gaskets, and construction with pentaborane. In particular, knowledge was required of the resistance to possible chemical attack of aluminum foil and 24S-T and 17S-T aluminum, 50-50 tin-lead solder, several natural and synthetic

rubbers, fluorinated polymers, asbestos and cellulose fibers, and graphite packing.

Although tests of the compatibility of pentaborane with some of the materials had been reported (refs. 1, 2, and 3), additional information was desired because of the short test times and uncertain temperature conditions involved in the reported work. A study was therefore undertaken to test the compatibility of metals and sealing materials with pentaborane under conditions simulating those which might be encountered in an actual use. Failure of any of the materials of construction can not be tolerated because of the extreme toxicity (refs. 1, 3, 4, and 5) of pentaborane, and its sensitivity to moisture and air which might result in explosive reactions.

The metals were tested in the liquid and vapor of pentaborane for about 7 hours in the range -30° to -50° F, at 90° F, and for 28 days at 70° F. Samples of the materials used for seals and gaskets were totally immersed in pentaborane for 7 days. Results were evaluated by comparing physical data taken on the treated samples with data on the original materials which were carried through the test procedure as blanks.

APPARATUS AND PROCEDURE

The apparatus for testing metal-pentaborane compatibility is shown in figure 1. It consisted of a series of 25- by 175-millimeter test tubes fused together by means of cross tubes, which were bent to make a unit small enough to fit a 2-quart Dewar flask. Long glass tubes of 6-millimeter outside diameter were attached to the apparatus to provide for transfer of the pentaborane from a vacuum system by methods described by Sanderson (ref. 6). When the liquid level was in the cross tubes, the specimens were kept separated from each other and yet were interconnected through a continuous liquid and vapor medium - a feature desirable for these tests in order to permit interaction among the samples if such action could occur.

The tests with the graphite packing and other sealing materials were performed in single test tubes to which 9-inch lengths of 6-millimeter tubing were fused after the sample had been inserted.

The aluminum alloy samples consisted of sheets of 1/32-inch 24S-T and 17S-T aluminum cut into 2- by 3/8-inch specimens. Holes drilled in the specimen identified the ends exposed to vapor, and notches distinguished between the types. These operations created stress areas which received special attention in the analysis of results. The foil was folded to test size from a 4- by 3/8-inch strip and the solder was cut to length from a commercial coil of 50-50 tin-lead solder (fig. 1).

Before being placed in the test apparatus, the metal specimens were washed with pumice soap, rinsed in absolute alcohol, and dried. One side of the aluminum was then scratched with a diamond pencil prior to weighing. The foil, however, was tested without this treatment. Three series of samples were prepared for tests in the range -30° to -50° F, at 70° F, and at 90° F.

The materials used for seals and gaskets were cut to size from 4- by 4-inch sample sheets, and test sections were made by two cuts of a die 2 inches in diameter which produced a test piece with an hour-glass profile. The graphite packing, which had already been in service but not exposed to fuel, was broken into two pieces, one of which was used for the test.

The metal samples were weighed, placed in the apparatus, and sealed in by fusing the glass. The apparatus was then attached to a vacuum system by means of 1/4-inch pressure tubing. Pentaborane from a tank was vaporized into a trap in the vacuum system. It was purified superficially by pumping away all components appreciably volatile at -78° C until successive freezing and melting produced no change in the vapor pressure at the freezing point. The pentaborane was then transferred by vaporization to the test apparatus, which was held at liquid nitrogen temperature. Some hydrogen was evolved when the solid pentaborane melted, but this was pumped off before the final seal was made on the introduction tube. Blank samples of each metal were sealed in individual tubes without pentaborane or air. The apparatus was then subjected to test conditions of -30° to -50° F for 7 hours, 70° F for 28 days, and 90° F for $6\frac{1}{2}$ hours.

When the test was completed, the apparatus was cooled in liquid nitrogen and a file mark was scratched on the introduction tube. The apparatus was attached to the vacuum system and a hose clamp placed on the pressure tubing below the scratch mark. Bending the rubber tubing caused the glass tip of the apparatus to break, thus exposing the pentaborane to the vacuum and permitting it to be pumped into a removable trap. After the pentaborane was removed, the samples were recovered by breaking the tips of the tubes holding them. When the apparatus was opened, the specimens acquired a coating of white oxide because of exposure to air prior to weighing. After being weighed, the specimens were examined under a metallurgical microscope for evidence of corrosion or other change.

Single tubes containing seal and gasket materials were prepared similarly, except that the specimens were completely immersed in liquid. The sample tubes stood for 7 days before they were opened and the samples inspected. Measurements were also made on the original material for comparative purposes.

CO-1 back

The specimens remained in the vacuum system for at least 15 minutes after visible liquid had evaporated. Removal of volatile matter was aided by application of infrared heat. Helium was then admitted to the system and the sample was removed by breaking the tube. In several instances, exposure to air resulted in ignition and loss of the specimen.

RESULTS AND DISCUSSION

Metals

Figure 2 shows the apparatus at the completion of the test run at -30° to -50° F. The apparatus was initially clear but became fogged after being exposed to air overnight. The results of this test are shown in table I. In general, the blanks lost 0.2 to 0.4 milligram during the test. This loss could be the result of the removal of adsorbed moisture and gases by heat radiated to the sample during the sealing process. The test samples gained 0.1 to 0.2 milligram because of an oxide film which was at first not removed. This visible deposit weighed about 0.3 milligram, as shown by the check weighing of sample number 13. In general, the magnitude of the differences before and after test was similar to the weighing error. It is therefore believed that no significant differences existed between treated and blank samples.

The data for the test at 90° F are given in table II. Figure 3 shows the apparatus before the samples were removed. The deposit on the apparatus was also caused by exposure to air overnight. It is evident from figure 3 that a greater decomposition of pentaborane occurred at the higher temperature. A larger amount of nonvolatile matter was produced, which oxidized to the light and dark colored deposits shown in the photograph. Small amounts of straw-colored, syrupy liquid were visible when the pentaborane was pumped away. Concurrent tests run on the thermal stability of pentaborane under similar conditions showed that the test samples exerted no observable catalytic effect on this decomposition. The amount of nonvolatile matter adhering to the specimens is indicated by weighings made before and after the sample was washed in water, in which the residue was completely soluble. The differences between weights of exposed and unexposed specimens is believed to be insignificant.

Figure 4 shows the apparatus at the conclusion of the test run at 70° F for 28 days. Special precautions were taken to avoid exposure of the apparatus to air. This view shows the nature of the residue left when the volatile matter had been removed from the system. Table III shows the results of the test after the samples were washed to remove the residue and dried. The changes in weight, for the purposes of the test, were believed to be insignificant.

At the conclusion of the experiments, the test specimens were submitted to a metallographer for microscopic analysis. No differences

between test samples and blanks and no evidence of action or corrosion could be detected in any part of the specimens.

Seal and Gasket Materials

The seal and gasket materials were removed from the test units after 7 days. The results are discussed for each specimen individually, and the data are summarized in table IV.

Buna rubber was so severely cracked and embrittled after the pentaborane had been removed that it could not be tested for tensile strength. The jaws of the testing machine crumbled the specimen as they were tightened. The weight of the sample increased 0.1816 gram or 16.6 percent. Since the outer surfaces of the specimen were clean (no oxide film) and dry, this increase in weight evidently represented pentaborane soaked into the pores. The weight continuously decreased during weighing, indicating vaporization of the pentaborane, but no oxide film formed. This observation is in accordance with another study completed recently at this laboratory in which it was concluded that the liquid but not the vapor of pentaborane reacts spontaneously with air.

Butyl rubber remained flexible enough for a tensile test in which it showed an 82.2-percent loss in its original tensile strength. Three peculiarities were manifested during this test. First, because the increase in tension was not quite uniform, the sample broke while under tension but not while undergoing a change in tension. The lever increasing the tension had momentarily stopped. Second, the sample broke at the wide end of the test specimen near the jaws of the machine and not at the narrow test section. Inspection at the break showed a small volume where an incipient blister had formed. Disrupted fibers in this area allowed liquid to collect and weakened the structure sufficiently to cause rupture of the sample at this point rather than at the test section. Third, while the sample was being returned in a closed tube from the tensile measuring area, an explosion occurred in the tube. The stopper was blown out but no fire ensued. Thereafter, the samples were not stoppered.

GR-S rubber gained 14.9 percent in weight, 10.7 percent in width, and 8.5 percent in thickness, and lost 91.9 percent of its original tensile strength. Before the pentaborane was pumped away, it exhibited a yellow-green fluoresence; and at the end of the removel operation, a yellow viscous syrup remained on the tube. This seemed insoluble in water, but effervesced quietly when alcohol was added. The sample was brittle and had hardened before removal from the tube.

Natural rubber seemed to retain its resiliency. One end ignited when the sample was removed from the tube, but the fire was extinguished.

Enough material was left for a tensile test. The sample parted at the wide section next to the jaws of the machine to give a loss of 97.7 percent of its original tensile strength.

Neoprene was flexible at the end of the test, but underwent a dimensional increase of about 10 percent which would correspond to a weight gain of about 15 percent. It broke under a stress of 32.2 pounds per square inch after stretching 5/16 inch. The break during test occurred at the thickened section next to the test section. The loss was 84.2 percent of its original tensile strength.

Silicone rubber also appeared flexible and not embrittled by the test. This sample seemed to have swelled the most of the specimens. It was very difficult to remove all the pentaborane from this sample. After the pentaborane was pumped away as described, helium was purged through the apparatus four times. A short flash occurred when the pressure tubing was removed, so the sample tube was plunged into dry ice. When the tube was removed from the dry ice, a large amount of solid had frozen onto the wall; the vacuum system was therefore reconnected. Evacuation then proceeded and the material which had oozed out of the specimen during freezing was pumped away. The sample was removed, but it ignited upon exposure to air and was consumed.

To avoid loss of the Thiokol rubber sample, JP-4 was added to the specimen tube while it was still under vacuum. Slight effervescence occurred, indicating some reaction with water. After the pentaborane was extracted, the tube was broken and the sample washed with acetone to remove JP-4. A final stress test could not be made because this specimen had broken into two pieces during the evacuation process. The gain in weight after extraction of the specimen with JP-4 was 10 percent.

The Teflon sample gained 0.6 milligram and showed a change in ultimate strength from 6340 pounds per square inch for the untreated sample to 3382 pounds per square inch after exposure to pentaborane. No change occurred in length, width, or thickness of the sample. The loss in ultimate strength may be apparent rather than real because the operator of the testing machine was required to stop to adjust the gripping jaws after the test had begun.

Kel-F showed a weight loss of 1.5 milligrams and changed in ultimate strength from 5890 pounds per square inch to 5412 pounds per square inch (8.1-percent loss). However, compared with Teflon, which underwent a 37.5-percent increase in length before breaking, the Kel-F broke when stretched 4.7 percent over its original length. The samples are shown in figure 5 before and after testing.

The graphite-impregnated asbestos specimen broke at the test section during exposure. The edges of the sample frayed somewhat, but the pentaborane remained clear. After the pentaborane was pumped away, JP-4 was

added. The tube was broken and the sample was washed in acetone and dried. No change in appearance, texture, or strength of the sample was discernible at the completion of the test.

Asbestos paper gained 4.3 percent in weight under the same treatment. Not enough material was available for a tensile test. No change, however, was apparent.

Vellumoid did not undergo any change in tensile strength or dimensions during the test, but gained 8.3 percent in weight.

The sample of graphite packing left a flocculent precipitate of flat platelets in the liquid, which turned yellow. This was the only sample of gasket material which showed a weight loss during the test. It amounted to 1.1 percent.

CONCLUSIONS

Results obtained when pentaborane was tested for compatibility with selected samples of aluminum, tin-lead solder, natural and synthetic rubbers, and asbestos and graphite sealing materials led to the following general conclusions:

1. Aluminum grades 24S-T and 17S-T, aluminum foil, and 50-50 tin-lead solder are satisfactory materials for use with pentaborane at temperatures to 90° F.

2. Teflon and Kel-F show no dimensional or physical change after exposure. These materials should be very useful with pentaborane.

3. Natural and synthetic rubbers absorb pentaborane estimated to amount to 50 percent of their own weight. When 1/16-inch samples are subjected to a high vacuum for about 15 minutes, 10 to 30 percent by weight is retained in the pores of the material.

4. Those natural and synthetic rubbers which are not embrittled under the test conditions, lose about 90 percent of their tensile strength after 7 days at 70° F in pentaborane.

5. The absorption of pentaborane disrupts the rubber structure so that the weakest area is not at the designed test section, but at a point where deterioration has caused an incipient blister.

6. Paper and asbestos based materials are satisfactory as gaskets under conditions of this test.

7. Graphite packing appears satisfactory, but pentaborane may extract the materials used as a binder.

8. Comparison of the amount of pentaborane residues obtained during these tests with other data obtained when pentaborane from the same source was exposed to heat alone shows qualitatively that mutual contact of the samples and the pentaborane has little effect on the decomposition of pentaborane.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, May 17, 1954

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TABLE I. - COMPATIBILITY TEST RESULTS

[Conditions: -30° to -50° F; 7 hours.]

Sample number	Material	Weight before test, g	Weight after test[a], g	Difference, mg
3	Al 24S-T	1.3291	1.3293	0.2
4	Al 24S-T	1.3371	1.3372	.1
12	Al 17S-T	.9808	.9809	.1
13	Al 17S-T	.9942	.9944	.2
			[b].9941	-.1
9	Al 24S-T (blank)	1.3317	1.3314	-.3
9	Al 17S-T (blank)	.9840	.9837	-.3
17	Al foil	.0884	.0884	0
20	50-50 Sn-Pb solder	3.3129	3.3131	.2
24	Al foil (blank)	.0797	.0793	-.4
24	50-50 Sn-Pb solder (blank)	3.2710	3.2708	-.2

[a]With deposit.
[b]Visible deposit removed.

TABLE II. - COMPATIBILITY TEST RESULTS

[Conditions: 90° F; $6\frac{1}{2}$ hours.]

Sample number	Material	Weight before test, g	Weight after test, g	Difference, mg
7	Al 24S-T (blank)	1.3338	1.3335	-0.3
7	Al 17S-T (blank)	.9709	.9705	-.4
22	Al foil (blank)	.0920	.0918	-.2
22	50-50 Sn-Pb solder (blank)	3.2618	3.2616	-.2
1	Al 24S-T	1.2796	[a]1.2839	4.3
			[b]1.2797	.1
2	Al 24S-T	1.3007	[a]1.3082	7.5
			[b]1.3008	.1
10	Al 17S-T	.9909	[a]1.0098	18.9
			[b].9908	-.1
11	Al 17S-T	.9898	[a].9965	6.7
			[b].9896	-.2
16	Al foil	.0841	[a].0921	8.0
			[b].0837	-.4
19	50-50 Sn-Pb solder	3.2400	[a]3.2425	2.5
			[b]3.2395	-.5

[a]With deposit.
[b]Without deposit.

CO-2

TABLE III. - COMPATIBILITY TEST RESULTS

[Conditions: 70° F; 28 days.]

Sample number	Material	Weight before test, g	Weight after test[a], g	Difference, mg
8	Al 17S-T (blank)	0.9830	0.9825	-0.5
8	Al 24S-T (blank)	1.3289	1.3286	-.3
23	Al foil (blank)	.0791	.0781	-1.0
23	50-50 Sn-Pb solder (blank)	3.2658	3.2656	-.2
5	Al 24S-T	1.3369	1.3365	-.4
6	Al 24S-T	1.2723	1.2718	-.5
14	Al 17S-T	.9924	.9920	-.4
15	Al 17S-T	.9830	.9827	-.3
18	Al foil	.0932	.0927	-.5
21	50-50 Sn-Pb solder	3.3768	3.3757	-1.1

[a]All deposits removed.

TABLE IV. - COMPATIBILITY TEST RESULTS

[Conditions: 70° F; 7 days.]

Material	Original tensile strength[a], psi	Final tensile strength[a], psi	Tensile strength loss, percent	Original stretch, in.	Final stretch, in.	Weight increase, percent	Change in width, percent	Change in thickness, percent
Rubbers:								
Buna	2550	------	----	9.8	---	16.6	----	----
Butyl	1790	318.0	82.2	7.8	1.4	30.7	----	----
GR-S	3070	250.0	91.9	4.8	.5	14.9	10.7	8.5
Natural	2700	63.5	97.7	7.1	.5	(b)	14.6	14.3
Neoprene	2040	322.2	84.2	7.6	1.3	----	8.8	9.2
Silicone[c]	491	------	----	1.9	---	----	----	----
Thiokol	976	(d)	----	3.1	---	10.1	----	----
Graphite asbestos	1470	(d)	----	---	---	0	0	0
Asbestos paper	----	------	----	---	---	4.3	0	0
Vellumoid	6060	6000.0	No change	---	---	8.3	0	0
Graphite packing	----	------	----	---	---	-1.1	0	0
Kel-F	5890	5412.0	8.1	---	.1	0	0	0
Teflon	6340	3382.0	[e]46.7	---	2.2	0	0	0

[a]Ultimate strength.
[b]Small fire occurred.
[c]Ignited.
[d]Broke during test.
[e]See RESULTS AND DISCUSSION for explanation of this value.

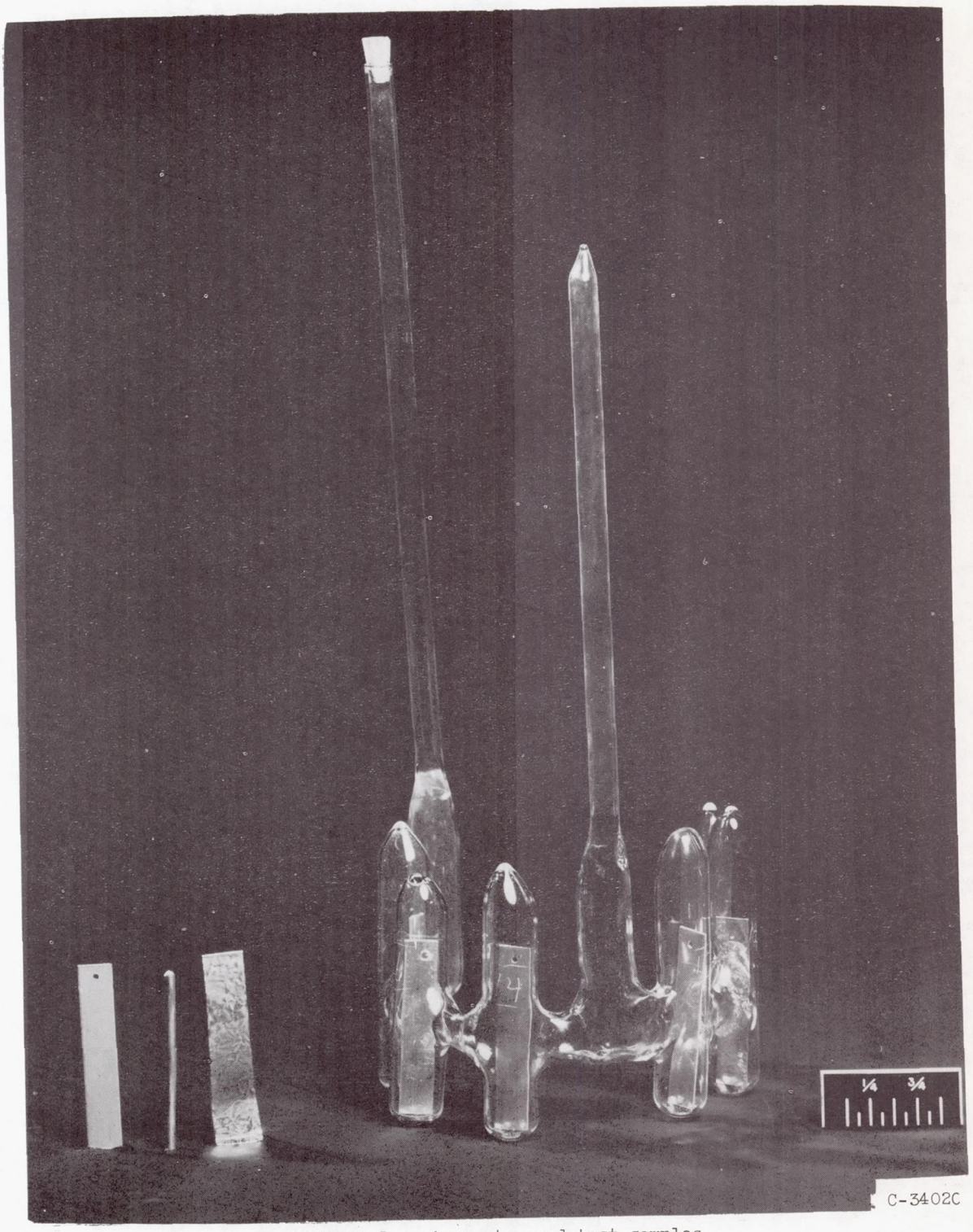

Figure 1. - Apparatus and test samples.

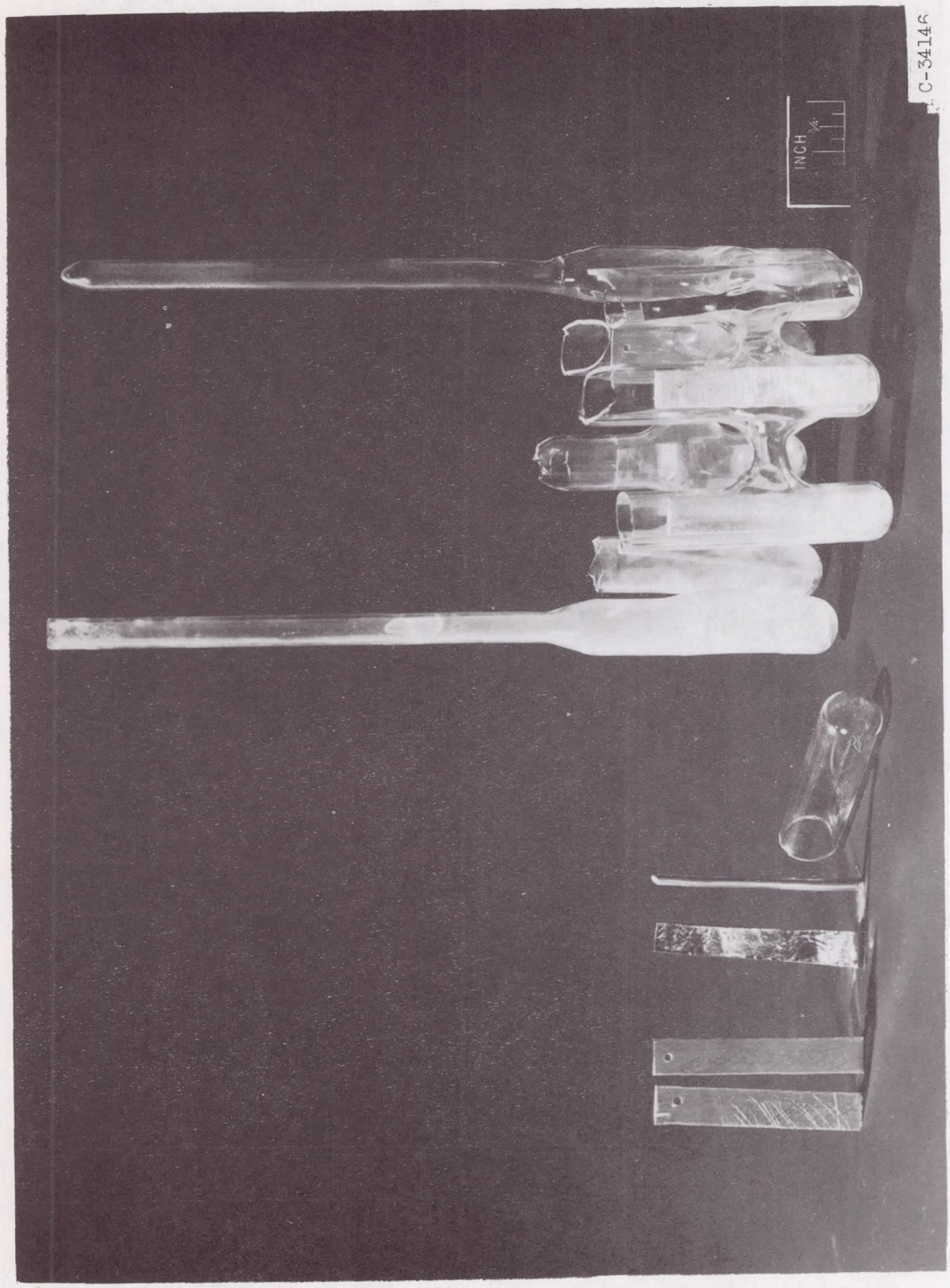

Figure 2. - Test in range -30° to -50° F, after 24 hours.

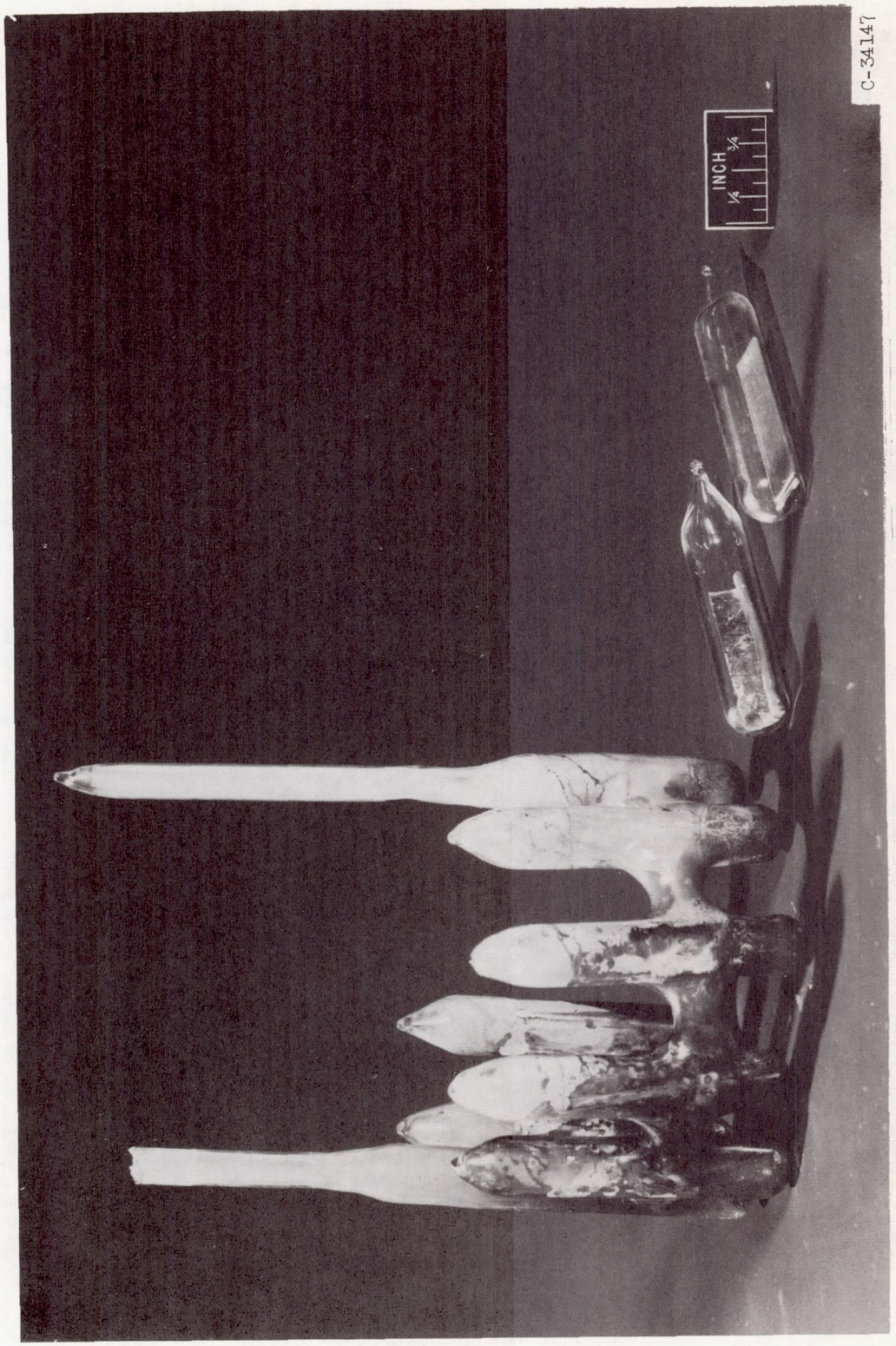

Figure 3. - Test at 90° F, after 24 hours.

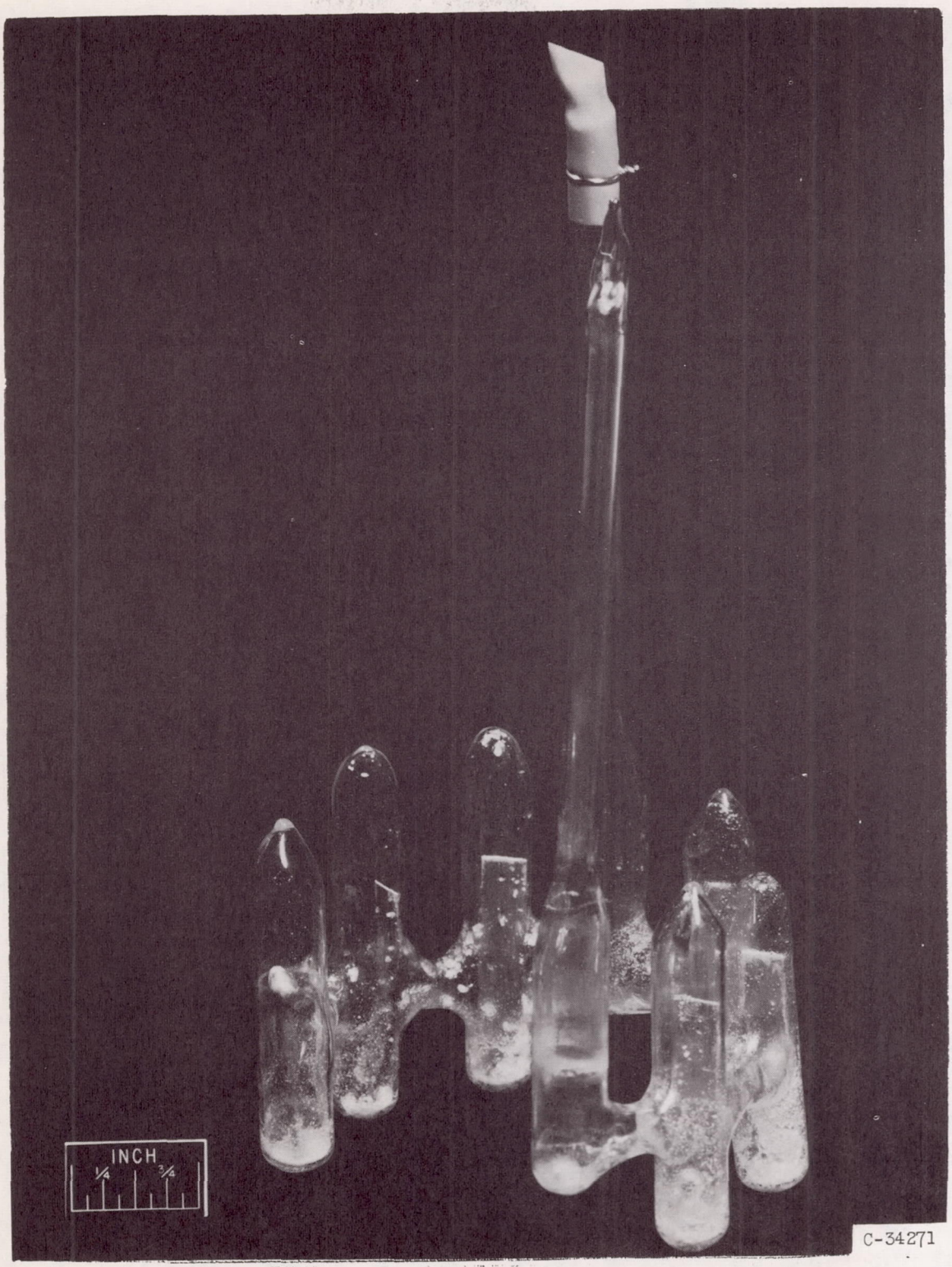

Figure 4. - Test at 70° F, after pentaborane was pumped away.

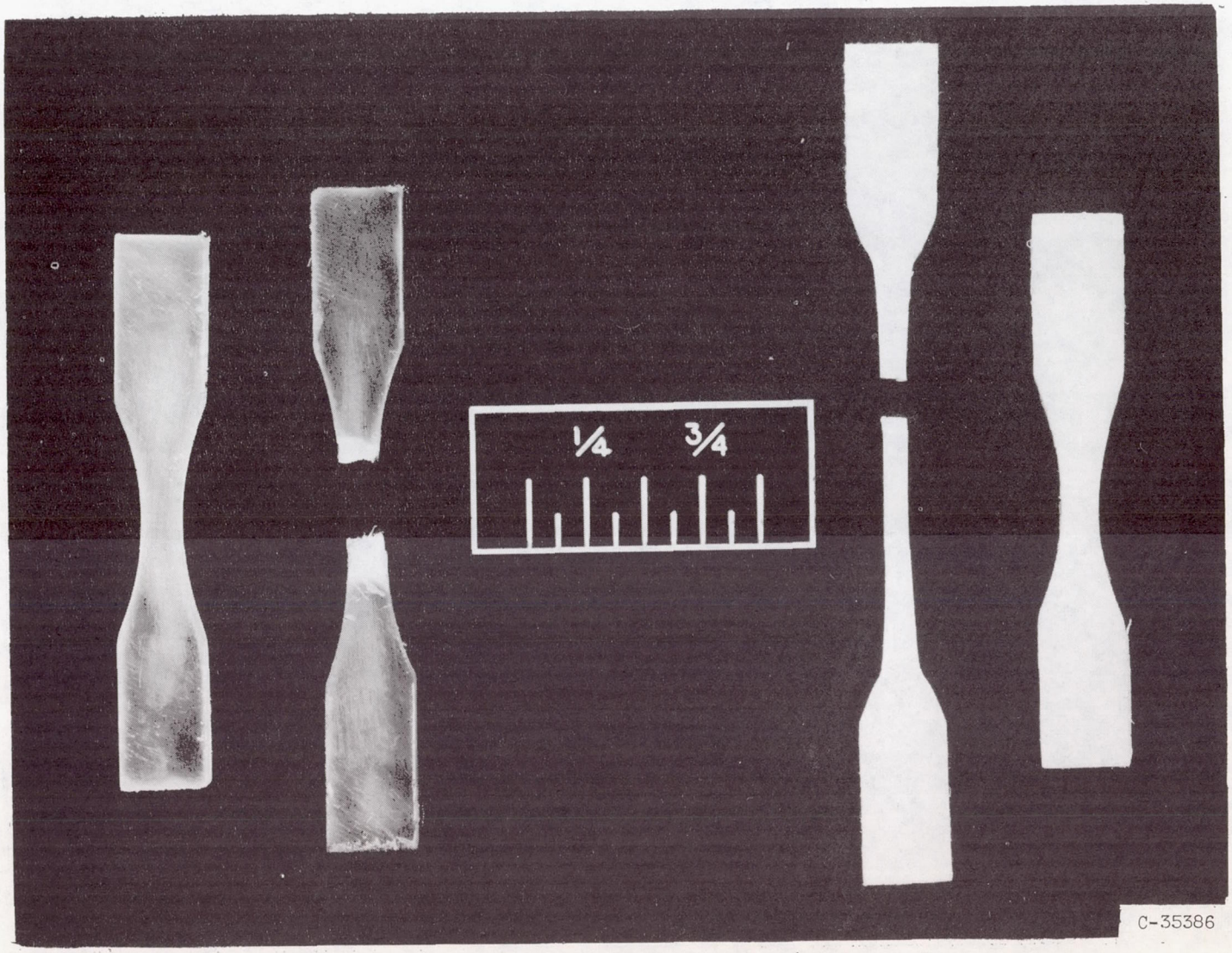

Figure 5. - Samples of Teflon (right) and Kel-F (left) before and after test.